



Effect of processing variables and bulk composition on the surface composition of spray dried powders of a model food system



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ABSTRACT

The surface compositions of food powders created from spray drying solutions containing various ratios of sodium caseinate, maltodextrin and soya oil have been analysed by Electron Spectroscopy for Chemical Analysis. The results show significant enrichment of oil at the surface of particles compared to the bulk phase and, when the non-oil components only are considered, a significant surface enrichment of sodium caseinate also. The degree of surface enrichment of both oil and sodium caseinate was found to increase with decreasing bulk levels of the respective components. Surface enrichment of oil was also affected by processing conditions (emulsion drop size and drying temperature), but surface enrichment of sodium caseinate was relatively insensitive to these. The presence of “pock marks” on the particle surfaces strongly suggests that the surface oil was caused by rupturing of emulsion droplets at the surface as the surrounding matrix contracts and hardens.

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1. Introduction

Food powders are typically prepared from solutions, suspensions or emulsions by spray drying. Spray drying is a rapid rate process, yet observations of particle surfaces have revealed that surface compositions are significantly different to the bulk composition of the powder (Adhikari et al., 2009; Fäldt et al., 1993; Fäldt and Bergensstahl, 1996a; Jayasundera et al., 2009; Kim et al., 2002, 2009a,b,c; Vignolles et al., 2007). There is also evidence that spray drying of emulsions causes coalescence of fat globules (Ye et al., 2007).

The surface of a powder is arguably the most important part of a food particle, because it is the surface that interacts with the external environment. Surface character may affect dissolution, reactivity, diffusivity, or the relative importance of the various surface forces. These have process implications such as better powder handling, powder storage, wettability, environmental equilibrium and shelf life. The presence of fat at the surface is particularly known to markedly compromise wettability (a key parameter for most powder applications), flowability and storage stability (from fat oxidation) (Vignolles et al., 2007). There are also product quality

factors that influence the delivery mechanism or have sales advantages. These include improved appearance, hydrophobicity, swelling, taste, controlled release kinetics, sensitivity to release environment, or human response factors such as optimal digestive uptake, satiety, probiotic viability, enteric coatings and making substances hypo-allergenic. These might involve scenarios, such as fried potato snack flavourings, where the material is ingested in its dry powdered form.

Surfaces are described by their character and integrity, where character is the composition and morphology of the topmost visible surface, and integrity is the mechanical strength and resistive capacity of the surface and near surface regions. Particle performance is acutely dependent on its surface character and integrity, in a myriad of ways. Surface composition defines physiochemical behaviours such as adsorption, glass transition, polarity, reactivity, dissolution, swelling or charge carrying capacity. The morphology defines shape and size which directly affects flowability and dispersibility. When considering integrity, the mechanical strength may be adequate to survive the processing conditions but, in its development, may cause rupture of encapsulated fat globules releasing free fat or, alternately, the resistive properties may be poor if the particle is porous.

Various industries wish to develop products that have both discernible benefit and attract premium prices, which is particularly

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true for commodity food powders because of the huge volumes involved (dairy as an example), but the margins do not allow additional processing steps to be entertained. It is thus useful to investigate avenues for tailoring surface character and integrity using only the means that could readily be employed by industry: namely, by manipulating the formulation, the process equipment design and the drying environment.

While stickiness is the principle processing issue, there is a growing awareness of the need to quantify and manipulate the surface composition of food emulsions when spray dried. The review of Jayasundera et al. (2009) concluded that, while there is a rich and growing literature on quantifying surface and bulk compositions, there is a dearth of information on the mechanisms responsible for the differences between bulk and surface compositions. Mechanisms have been proposed recently for milk powders by Kim et al. (2009a,b,c). In these papers, the surface compositions of spray dried milk powders were studied for a range of milk compositions (whole milk, skim milk, instant whole milk, cream), the spray drying conditions (air inlet and outlet temperatures) and the changes that occur with storage. They found that the surface composition is determined during spray drying rather than in any of the other processing steps (e.g. conditioning fluid bed dryers or packaging). Interestingly, for instant whole milk powder, the soy lecithin (added as a natural surfactant) could not be detected at the powder surface. They explained this lack of detection as partly due to the small amount applied, 0.1–0.4%, and also that the lecithin was sprayed onto the powder dissolved in anhydrous milk fat. They also found that fat was present at the surfaces of the powders in far greater proportion than present in the bulk, confirming observations by Fäldt and Bergenstahl (1996a). Over long storage times they found no effective change in the surface composition as measured by Electron Spectroscopy for Chemical Analysis (ESCA). This is because the lactose remained in the amorphous form due to the presence of a desiccant; however, after washing with organic solvents, some release of the low melting triglycerides was detected. Humid conditions were investigated by Fäldt and Bergenstahl (1996b) who observed the release of free fat to the surface.

Kim et al. (2009b) proposed mechanisms for the formation of the surface composition as follows. Citing earlier work (Dombrowski and Fraser, 1954; Zakarlan and King, 1982), they proposed that the instabilities that form droplets begin at the interface between the oil and the continuous aqueous phase. This then results in the surfaces of the newly formed spray droplets being well represented by emulsified oil, even before drying commences. Subsequent drying and the associated mass transfer kinetics promote further segregation. In the continuous phase, water moves along a concentration gradient towards the surface. Being a small molecule, water moves relatively quickly and carries with it large molecules which cannot diffuse as quickly in the opposite direction. Thus molecules may become segregated depending on their molecular weight. The emulsified oil and protein micelles could also be carried along in the convective flux of water moving toward the surface. This movement occurs until the continuous phase becomes relatively immobile, and thereafter the remaining continuous phase shrinks as the water leaves as vapour. Kim et al. (2009b) warn that the concentration gradients may not be observed if the drying temperature is high and the droplets rapidly form a crust. In this case, the system may be quenched, where the components are relatively evenly distributed.

To further investigate this phenomenon, a series of experiments were performed to examine the surface compositions when spray drying a model food aqueous emulsion containing a protein (sodium caseinate), a carbohydrate (maltodextrin) and a fat (soya oil) in a matrix of compositions, so that the effect of different components on surface enrichment can be studied over a wide range of compositions.

The powders used here are not milk powders as used by Kim et al. (2009a,b,c); rather, they are more similar to those of Fäldt and Bergenstahl (1996a,b). Instead of lactose, maltodextrin DE10 is used as the carbohydrate, which has a higher glass transition temperature. Soya oil is used here instead of milk fat, which has a wide range of triacylglycerol chain lengths. Rather than using the whole range of milk proteins, this work selects the food grade refined form of the most significant protein, sodium caseinate. It is also known to be a better encapsulant than whey protein (Fäldt and Bergenstahl, 1996b) although is known to form interfaces that are more rigid and less elastic (Jayasundera et al., 2009). Therefore, sodium caseinate will provide a realistic model food system with which to observe surface composition and the surface release of oil. Thus, these differences mean the system used here is compositionally simple. It also allows flexibility to vary the emulsion composition.

2. Materials and methods

Emulsions were prepared from three ingredients: soya oil (Soyola, Kore S.A., Koropi, Greece), maltodextrin DE10 (Maltrin M100, Paroxite Ltd., Macclesfield, UK) and sodium caseinate. Two grades of sodium caseinate were compared – food industry (92.5% protein) grade (Adpro S, Adams Food Ingredients, Leek, UK), and a purer (99% sodium caseinate) grade (Sigma, Poole, UK).

The ingredients were mixed according to Table 1, which yielded the simple water-free composition map shown in Fig. 1. The ingredients were stored in airtight containers and weighed to within 0.1 g into either a 4 or 8 L container without baffles, depending on the dilution required. The total water-free mass of the ingredients was 1200 g. The sodium caseinate content dominates the emulsion viscosity and so water was generally added to each mixture in the ratio of six parts water to one part of sodium caseinate, except occasionally when maltodextrin levels were high, which had a secondary effect on emulsion viscosity. These dilutions were performed to ensure that the emulsions could be delivered by the peristaltic pump to the top of the spray dryer.

2.1. Homogenisation

Emulsification/homogenisation was performed in a benchtop homogeniser (Ultra Turrax T-50, Ika-Werke GmbH, Staufen, Germany). This is a blade-in-cage assembly. The emulsion was formed by blending for 1 min at 3000 rpm, followed by 1 min at 7000 rpm, then 8 min at 10,000 rpm. At the lower speeds, a spatula was used to ensure lumps were circulated towards the contact zone and that no protein gel formed at the walls. The container used was a cylindrical bucket, without baffles. After blending, the free surface of the resulting emulsion had sheen, indicating it was well homogenised. Due to the high energy input of the high-shear homogeniser, the temperature of this mixture could exceed 55 °C. The mixture was

Table 1
Compositions of feed solutions tested (wt.%).

Feed solution	Sodium caseinate	Maltodextrin	Soya oil	Water
A	11.9	4.8	11.9	71.4
B	11.9	11.9	4.8	71.4
C	8.3	20.8	20.8	50.0
D	8.3	8.3	33.3	50.0
E	8.3	33.3	8.3	50.0
F	12.1	3.0	3.0	81.8
G	12.5	0.0	12.5	75.0
H	11.1	11.1	0.0	77.8
J	13.6	4.5	0.0	81.8
K	10.0	30.0	0.0	60.0
L	8.3	8.3	8.3	75.0
M	0.0	50.0	0.0	50.0
N	14.3	0.0	0.0	85.7

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